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IS 12519 (1988): Zinc pyrophosphate for electroplating [CHD
5: Electroplating Chemicals and Photographic Materials]



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Indian Standard
**SPECIFICATION FOR
ZINC PYROPHOSPHATE FOR
ELECTROPLATING**

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Indian Standard

SPECIFICATION FOR ZINC PYROPHOSPHATE FOR ELECTROPLATING

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 21 October 1988, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 IS : 1880-1977* has already been published to serve the electroplating industry. Since phosphate plating and electrocoating have recently developed in electroplating industry, the Committee decided to formulate this standard.

0.3 Zinc pyrophosphate is used as an ingredient of electroplating baths for zinc and its various

alloys. The salt is also extensively used for phosphate plating (which was effective corrosion resistance) and electroplating on various metals and its alloys.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Specification for zinc oxide and zinc salts for electroplating (first revision).

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and tests for zinc pyrophosphate electroplating.

2. REQUIREMENTS

2.1 Zinc pyrophosphate shall be in the form of a white powder, insoluble in water but soluble in acids and alkalies shall correspond essentially to the formula $Zn_2P_2O_7$.

2.2 The material shall also comply with the requirements given in Table 1, when tested in accordance with the methods prescribed in Appendix A. References to relevant clauses of Appendix A given are in col 4 of Table 1.

multiwall paper bags/drums or polyethylene bags not exceeding 50 kg in mass when packed.

3.2 Marking

3.2.1 The containers shall be marked with the following particulars:

- a) Name of the product;
- b) Name of the manufacturer and/or his recognized trade-mark, if any;
- c) Net mass of the product;
- d) Date of manufacture; and
- e) Batch/code number.

3.2.2 The containers may also be marked with the Standard Mark.

**TABLE 1 REQUIREMENTS FOR ZINC
PYROPHOSPHATE FOR ELECTROPLATING**

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (Ref TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Zinc pyrophosphate (as $Zn_2P_2O_7$), percent by mass, <i>Min</i>	98.0	A-2
ii)	Chloride (as Cl), percent by mass, <i>Max</i>	0.05	A-3
iii)	Sulphate (as SO_4), percent by mass, <i>Max</i>	0.01	A-4
iv)	Iron (as Fe), percent by mass, <i>Max</i>	0.005	A-5
v)	Heavy metals (as Pb), <i>Max</i>	0.001	A-6

3. PACKING AND MARKING

3.1 Packing — The product shall be packed in

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 The method of drawing representative samples of the product and criteria for conformity of the material to the requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(Clause 2.2, and Table 1)

METHODS OF TEST FOR ZINC PYROPHOSPHATE FOR ELECTROPLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless otherwise specified, pure chemicals and distilled water (see IS : 1070-1977*) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF ZINC PYROPHOSPHATE

A-2.1 General — Zinc pyrophosphate is determined by means of redox titration of zinc ions in solution with a standard potassium ferrocyanide solution to form the very sparingly soluble potassium zinc ferrocyanide.

A-2.2 Reagents

A-2.2.1 0.05 N Potassium Ferrocyanide Standard Solution — Weigh accurately 21.12 g of pure potassium ferrocyanide [$K_4Fe(CN)_6 \cdot 3H_2O$] and 0.3 g of potassium ferrocyanide in a 1-litre volumetric flask. Dissolve in distilled or conductivity water and make up to the mark.

A-2.2.2 Standard Zinc Solution (0.1 M) — Take 1.6345 g of pure zinc or 2.0345 g of ignited zinc oxide, dissolve in 10 ml of 20 percent (v/v) sulphuric acid and make up to 250 ml in a volumetric flask. One millilitre of this solution contains 6.538 mg of zinc.

A-2.2.3 Dilute Sulphuric Acid — 20 percent (v/v).

A-2.2.4 Ammonium Sulphate — solid.

A-2.2.5 1 Percent (m/v) solution of diphenylbenzidine in concentrated sulphuric acid.

A-2.3 Procedure — Pipette out 25 ml of standard solution of zinc into a 250 ml conical flask and add 50 ml of water, 25 ml of dilute sulphuric acid, 10 g of ammonium sulphate and not more than 3 drops of diphenylbenzidine indicator. Titrate the cold solution slowly and with vigorous shaking until the colour change from blue-violet to pale green is permanent. Calculate the volume of potassium ferrocyanide equivalent to 0.01 g of zinc.

NOTE — More than 3 drops of diphenylbenzidine indicator, if used, will give rise to low results.

A-2.3.1 Repeat the titration with 25 ml portions of the test solution prepared by dissolving 2.500 g of the material in 50-60 ml of water acidified with 1 ml of concentrated sulphuric acid, and making up to 250 ml.

A-2.4 Calculation

Zinc pyrophosphate as ($Zn_2P_2O_7$),
percent by mass = $\frac{2.3302 \times V_2}{V_1 \times M}$

where

V_1 = volume of potassium ferrocyanide solution equivalent to 0.01 g of zinc,

V_2 = volume of standard potassium ferrocyanide solution required for titration of the test solution, and

M = mass in g of the sample taken for the test.

A-3. DETERMINATION OF CHLORIDE

A-3.1 Reagents

A-3.1.1 Concentrated Nitric Acid — see IS : 264-1976*.

A-3.1.2 Silver Nitrate Solution — Approx 0.25 M or 3.4 percent (m/v) solution.

A-3.1.3 Standard Chloride Solution — Dissolve 0.824 g of dried sodium chloride or 1.051 g of potassium chloride in a 1-litre volumetric flask and make up to the mark. Pipette 10 ml of this aliquot into another 1 litre flask and dilute with water up to the mark. One millilitre of this dilute solution contains 0.005 mg of chloride (as Cl).

A-3.2 Procedure — Pipette 25 ml of the zinc pyrophosphate solution prepared for test (see A-2.3.1) in a Nessler cylinder, add 3 ml of concentrated nitric acid and 1 ml of silver nitrate solution. Dilute with water and mix well. Carry out a control test in another Nessler cylinder using 25 ml of standard chloride solution and the same quantities of the other reagents.

A-3.2.1 The material shall be taken as not having exceeded the limit prescribed in Table 1, if the turbidity produced with the material is not greater than that produced in the control test.

A-4. DETERMINATION OF SULPHATE

A-4.1 Reagents

A-4.1.1 Dilute Hydrochloric Acid — 20 percent (v/v).

A-4.1.2 Barium Chloride Solution — approximately 0.2 M or 5 percent (m/v).

A-4.2 Procedure — Dissolve 5 g of the material in a mixture of 25 ml of water and 10 ml of dilute hydrochloric acid in a 100 ml glass beaker and boil gently for 5 minutes. Cool, dilute to 50 ml, add 2 ml of barium chloride solution and transfer the material quantitatively to Nessler cylinder for comparison of turbidity or

*Specification for water for general laboratory use (second revision).

*Specification for nitric acid (second revision).

opalescence. Allow to stand for 2 hours. The turbidity produced shall not exceed that obtained with a similarly treated 1.00 ml standard solution of sodium sulphate prepared by dissolution of 0.7393 g of the anhydrous salt in water in a 1 litre volumetric flask.

A-5. DETERMINATION OF IRON

A-5.1 Reagents

A-5.1.1 Dilute Hydrochloric Acid — 20 percent (v/v).

A-5.1.2 Dilute Ammonia Solution — 50 percent (v/v).

A-5.1.3 Hydroxylamine Hydrochloride — 10 percent (m/v).

A-5.1.4 Sodium Acetate — 10 percent (m/v).

A-5.1.5 Bathophenanthroline — 0.0825 percent (m/v) solution in ethanol.

A-5.1.6 Disodium Salt of EDTA

A-5.1.7 Standard Iron Solution — Dissolve 7.0215 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] or 4.9781 g of ferrous sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$) with 5 ml of 20 percent (v/v) sulphuric acid solution in a 1000 ml volumetric flask. Take 10 ml aliquot of the same and dilute to 1000 ml in another 1000 ml volumetric flask. One millilitre of this diluted solution contains 0.01 mg of iron (as Fe).

A-5.2 Procedure — Weigh out exactly 2 g of the material into a 250 ml volumetric flask and add 100 ml of water followed by 10 ml of dilute hydrochloric acid. Pipette out 25 ml of the made-up solution into a 250 ml glass beaker and adjust its pH to 4-5 carefully by controlled addition of dilute ammonia. Add 1 ml of hydroxylamine hydrochloride, 2 ml of sodium acetate and about 0.3 g of disodium salt of EDTA which can form a soluble complex with zinc ions. Make up to 50 ml in a volumetric flask and mix. Add 0.5 ml of bathophenanthroline and read at 530 nm against a reagent blank, making use of a photoelectric colorimeter or a spectrophotometer. Determine iron content of the test solution by reference to a calibration curve,

prepared with known quantities of the standard iron solution under similar conditions.

A-5.3 Calculation

$$\text{Iron (as Fe) content} = M \times 500$$

where

M = mass in g of iron in the aliquot solution taken for the test.

A-6. DETERMINATION OF HEAVY METALS PRECIPITATED BY HYDROGEN SULPHIDE

A-6.1 Reagents

A-6.1.1 Dilute Hydrochloric Acid — 20 percent (v/v).

A-6.1.2 Dilute Ammonia — 25 percent (v/v).

A-6.1.3 Standard Lead Solution — Dissolve 0.1831 g of lead acetate trihydrate in water slightly acidified with acetic acid in 1-litre volumetric flask and make up the volume. 10 ml of this aliquot shall be further diluted to 1000 ml in another 1 litre flask. One millilitre of this diluted solution contains 0.01 mg of lead (as Pb).

A-6.1.4 Hydrogen Sulphide Gas — Produce in a Kipp's apparatus by the reaction of ferrous sulphide with 10 percent (v/v) sulphuric acid, and pass through water in a wash bottle.

A-6.2 Procedure — Dissolve exactly 5 g of the material in a mixture of 25 ml of water and 20 ml of dilute hydrochloric acid in a 250 ml beaker, boil gently for 5 minutes and cool. Transfer 10 ml of dilute ammonia solution to the beaker. Pass hydrogen sulphide gas for a few seconds. No turbidity shall be produced and any colour obtained not deeper than that obtained when, to a mixture of 1 ml of the standard lead acetate solution, a portion of the standard iron solution (1 ml = 0.01 mg Fe) containing the same quantity of iron as determined in test A-5 is added along with 40 ml of water and 10 ml of dilute ammonia solution and hydrogen sulphide passed through for a few seconds.

APPENDIX B

(Clause 4.1)

SAMPLING OF ZINC PYROPHOSPHATE FOR ELECTROPLATING

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test sample, the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.2 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.3 The samples shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.4 Each sample container shall be sealed air-tight after filling and marked with full

details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from a lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED

LOT SIZE (N)	NUMBER OF CONTAINERS TO BE SELECTED (n)
(1)	(2)
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

B-2.3 All the containers shall be selected at random and in order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLES AND REFEREE SAMPLES

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument, a small portion of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under 2 and shall not exceed 500 g.

B-3.1.2 Mix thoroughly all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 200 g. This composite sample shall be divided into three equal parts, one for the purchaser, another for the manufacturer and the third to be used as referee sample.

B-3.1.3 The remaining portions of the material from each container (after a small quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing not less than

50 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given under B-1.4. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the manufacturer and the third shall be used as the referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (see B-3.1.2) and a set of individual samples (see B-3.1.3) marked for this purpose. It shall also bear the seals of the purchaser and the manufacturer. These shall be kept at a place agreed to between the purchaser and the manufacturer and shall be used in case of dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Tests for determination of potassium pyrophosphate shall be conducted on each of the individual samples for all the grades.

B-4.2 Tests for the remaining characteristics shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Sample

B-5.1.1 For Zinc Pyrophosphate — The test results for zinc pyrophosphate shall be recorded, and the mean and range for these test results shall be calculated as follows:

Mean (\bar{X}) = Sum of the test results divided by the number of test results, and

Range (R) = The difference between the maximum and minimum values of the test results.

The value of expression ($\bar{X} - 0.6 R$) shall be calculated. If the value of this expression is greater than or equal to the limits specified in Table 1, the lot shall be declared to have satisfied the requirements for these characteristics.

B-5.1.1.1 In case of sample of size 10, the first five test results may be taken in one group and the next five in another group. R shall be calculated for each of the groups and the average value of the two shall be calculated as R . If $\bar{X} - 0.6 R$ is greater than or equal to the limits specified in Table 1, then the lot shall be declared to have satisfied the requirements for these characteristics.

B-5.2 For Composite Sample — The test results on the sample shall meet the corresponding requirements specified in Table 1.

B-5.3 A lot shall be declared as conforming to the specification if it satisfies the requirements for each of the characteristics listed in Table 1.

*Methods of random sampling.

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